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AN ASSESSMENT OF TEST ENVIRONMENTS FOR THE STRESS CORROSION CRACKING OF SMALL CALIBER BRASS CARTRIDGE CASES

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FOREWORD

The impetus given the search for a non-polluting, stress-corrosion cracking test for brass cartridge cases to replace the conventional mercurous nitrate test, developed from recent discussions among personnel of the Small Caliber Ammunitions Branch and the Metals Forming and Processing Branch. Precedent information was found insufficient to permit immediate substantiation of potentially useful alternates. A project was established (#F628098 (J74), AMCMS 4110.16.0217.8.06.02) for the purpose of accessing non-polluting substitute tests.

An earlier evaluative study was made by the Metals Forming and Processing Branch, using 5.56 mm brass cases subjected to ammonia vapor and to Mattsson's Solution (NH_4SO_4 + $CuSO_4$). The results indicated the need for further discrimination and evaluation of potentially substitutive methods. The Materials Application Branch, Materials Engineering Division, was asked to pursue the investigation and to expand it as necessary. Exploratory evaluations were performed to assess the merit of alternate methods and to recommend approaches more likely to be successful. This report treats that assessment.

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13. ABSTRACT

Non-polluting and non-toxic stress corrosion testing methods, applicable to brass cartridge cases, are evaluated as potential alternates for the mercurous nitrate method. In this assessment, rapidity and reliability of indicating stress corrosion susceptibility are important considerations. Included are: vapor tests - specimens over ammonium hydroxide, ammonium carbonate, or various organic amine liquids; solution tests - specimens immersed in ammonium hydroxide, devoid of or containing hydrogen peroxide, in amine liquids, or in Mattsson's solution.

Stressed or stress-relieved specimens are employed. The results of these experiments are compared to those of the mercurous nitrate test. Specific conclusions are rendered concerning the ammonia vapor and the Mattsson test methods, and a recommendation is made concerning modifying the Mattsson test method.

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INTRODUCTION

The mercurous nitrate test method long has been used for detecting stress corrosion cracking susceptibility of fabricated brass components.[1] It is a relatively simple and rapid method, and its effects with susceptible materials are quite dramatic. However, the danger of polluting the environment and the probability of mercury poisoning compels the search for an alternate, rapid test method.

It is well established that ammonia vapor will effectively crack stressed brass,[2] but where equivalent testing times are involved the ammonia vapor will not be as catastrophic as the mercurous solution. Nonetheless, it was viewed that ammonia might be further evaluated as an effective medium for revealing crack susceptibility of brass cartridge cases. Experiments performed with ammonia are described in this report.

Ammonia related compounds also are recognized to cause cracking in brass. For example, Rosenthal and Jamieson[3] demonstrated that vapors of methylamine, demethylamine, trimethylamine, ethylamine, analine, and triethanolamine will effect stress corrosion cracking after somewhat extended exposure periods. In this work, some amines were evaluated. Another ammonia derivative used in this study was ammonium carbonate. Experiments were conducted with brass cases exposed to vapors of that compound.

Mattsson[4] devised a solution environment for testing stressed brass, consisting of ammonium and copper sulfate alkalinized to pH 7.2 with ammonium hydroxide. Mattsson's procedure also was evaluated with respect to the mercurous nitrate test.

Applied anodic current was employed in some of the tests conducted in ammonia medium. The intent was to accentuate cracks formed and to shorten the testing time.

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[1] "Mercurous Nitrate Test for Copper and Copper Alloys", ASTM Committee B-5 on Cu and Cu Alloys, Designation B154-71, p. 291.

[2] G. Edmunds, E. A. Anderson, and R. K. Waring, Symposium on Stress-Corrosion Cracking of Metals (ASTM - AIME, 1944), 1945: New York (Amer. Inst. Min. Met. Eng.), p. 7.

[3] H. Rosenthal and A. L. Jamieson Trans. AIME, 156, 212 (1944).

[4] E. Mattsson, Electrochim Acta, 3, 279 (1961).

Conclusions regarding the ammonia vapor and the Mattsson test procedures, and a recommendation for further study of the Mattsson method are presented.

EXPERIMENTAL WORK

Materials

Brass cases, 5.56 mm., taken from the production line, were employed. These consisted of two separate groups: 1- cases not stress relief annealed, (also referred to as stressed or unannealed), after the necking-down operation, 2- cases annealed, (also referred to as stress relieved), after the forming operation.

Cleaning Procedure

Annealed and unannealed cartridge cases were cleaned in alkaline cleaner (METSO-200/H_2O, 120 g/l) at 160°F for 15 minutes, rinsed in water, dried and weighed. After pickling in 40 percent nitric acid for 15 seconds, the specimens were rinsed, dried and weighed again. Specimens which were tested in ammonia vapor, were wet with water immediately before exposure, since a thin film of water on the specimen is essential in this test.

Test Methods

Mercurous Nitrate Test

Cleaned specimens, annealed and unannealed, were immersed in mercurous nitrate solution (10.7 g/l cntng 10 ml of HNO_3) for 30 minutes. The specimens were then washed and wiped free of excess mercury. After weighing the specimens, the amalgamated mercury was evaporated off by setting the specimens on a hot plate. Specimens were again weighed and then examined visually and microscopically for stress corrosion cracks.

Ammonia Vapor Test

Cleaned specimens (annealed and unannealed) were exposed to ammonia vapor for 15, 20, and 30 minute periods.

The test apparatus consisted of a 200 ml wide-mouthed jar containing a 50 ml beaker. Ammonium hydroxide solution was placed in the beaker (ca. 40 ml) and the cartridge cases were positioned outside the beaker in the jar. The tests were timed from the moment the specimens were cleaned of tarnish in 40 percent acid, and examined visually and microscopically for cracks.

Ammonium Hydroxide and Hydrogen Peroxide Immersion Test

Approximately 5 ml of hydrogen peroxide (3 percent/volume) was added to 40 ml of 8.06 M ammonium hydroxide and cleaned specimens were submerged in the solution for one hour. The specimens then were withdrawn, cleaned of tarnish and examined for cracks.

Ammonium Carbonate Test

Cleaned unannealed specimens were placed in the apparatus described for the ammonium hydroxide test but containing 15 grams of ammonium carbonate. Half of the specimens were wet when placed in the test container while the other half were dry.

The specimens were exposed for periods of 30, 60, and 90 minutes. Following exposure, the specimens, which were untarnished, were examined visually and microscopically.

Mattsson's Solution

Mattsson's solution of pH 7.2 was prepared according to the ASTM Recommended Practice.[5] This consisted of 590.0 grams ammonium sulfate dissolved in four liters of water and 125.0 grams of copper sulfate dissolved in one liter of water. These solutions were then mixed and 71.0 ml ammonium hydroxide solution was added. The mixture was then diluted to ten liters.

After aging the solution for 96 hours, cleaned specimens

[5] ASTM G.01.06, Section 2, Task Group 14, "Recommended Practice for the Use of Mattsson's Solution of pH 7.2 to Evaluate Stress-Corrosion Cracking Susceptibility of Cu-Zn Alloys", Third Draft, April 1972.

(annealed and unannealed) were suspended in the solution by cotton thread. The suggested minimum ratio for volume of test solution and exposed surface area, 30 ml/cm^2, was complied with.

Both the annealed and unannealed specimens were immersed in the solution for one, two, and three-hour periods. After extracting the specimens and removing the tarnish, the specimens were examined for cracks.

Amine Vapor Test

About 40 ml of the following amines were placed in separate 50 ml beakers: diethylamine, 2-ethylhexylamine, cetyl-dimethylamine, 2-(2-aminoethylaminoethanol), tetraethylene pentamine, triethanolamine and tributylamine. Each was placed in a separate container, same as that used for the ammonium hydroxide vapor test. Cleaned, unannealed cases were then placed into the containers and the containers were capped. Annealed specimens were tested in diethylamine, 2-ethylhexylamine and cetyl dimethylamine vapor.

Specimens were removed from the containers after various time intervals and examined for stress corrosion cracks. The total time of exposure did not exceed 25 hours.

Amine Immersion Test

Cleaned, unannealed specimens were immersed in the following amines: diethylamine, 2-ethylhexylamine, and cetyl dimethylamine. Samples were immersed for 17 and 24 hours and then examined for stress corrosion cracking.

Ammonium Vapor and Anodic Current Test

Annealed and unannealed cases were exposed to ammonia vapor, and an anodic potential of either 3 or 6 volts was imposed providing a current of 800 or 3110 ma. In this experiment the specimens were over electrolyte solution consisting of 8.06 M ammonium hydroxide (90 percent/volume) and 1 M sodium chloride (10 percent/volume). Figure 1 is a sketch of the apparatus employed, showing electrical contact between the electrolyte solution and the specimen, achieved by means of a felt wick inserted in the mouth of the cartridge case.

After 20 minutes of exposure, the specimens were removed from the apparatus, and were cleaned of tarnish. The case specimens were then examined for cracks.

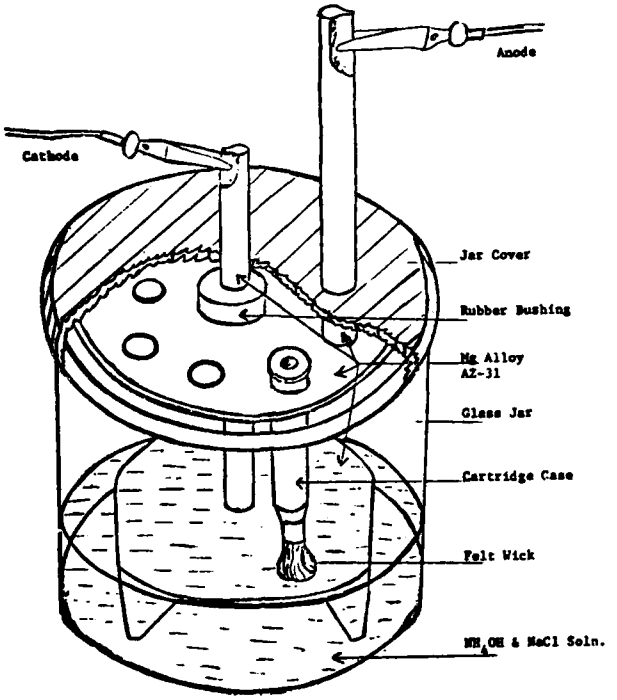


Figure 1. Anodic Current Apparatus

RESULTS

Mercurous Nitrate Test

Pronounced stress corrosion cracking of unannealed case specimens occurred with the mercurous nitrate solution. Examination of unannealed specimens after vaporizing the mercury, revealed severe cracking

in the neck area of each cartridge case. In no instance were stress corrosion cracks found in the body or base of the cases. The annealed specimens did not develop cracks after exposure to mercurous nitrate solution for the prescribed time (30 minutes). See Figure 2.

The cracks penetrated the entire thickness of the unannealed specimens, and in some instances the neck of the case was completely disengaged from the body.

Cracks could not be detected until the malgamared mercury had been evaporated. On removing the mercury from the specimens, cracks could be detected without the use of a microscope.

Ammonia Vapor Test

Dry or water-wet specimens exposed to vapor over 8.06 M ammonium hydroxide developed tarnish and fine cracks within a 30 minute period. These effects occurred more slowly with the originally dry specimens, on which a liquid film gradually developed. As a consequence, all subsequent tests were performed on wet specimens.

The susceptibility of unannealed and annealed cases to stress corrosion cracking in ammonium hydroxide vapor is shown in Tables I and II, respectively. All unannealed specimens exhibited cracks at 30 minutes, regardless of the concentration of the ammonium hydroxide solution. The annealed specimens exposed over solutions less than 2.4 M did not crack in 30 minutes. Cracks which resulted over the solution of highest ammonia concentration were superficial and did not penetrate the case wall. Cracks that developed in annealed cases could not be distinguished from those produced in unannealed specimens.

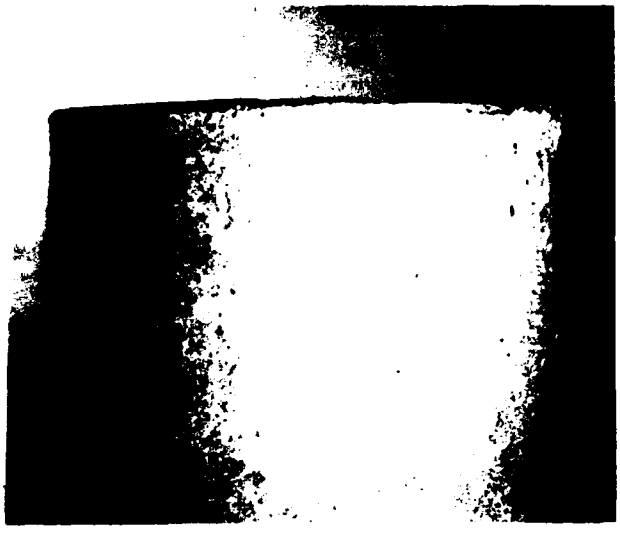
The cracks produced in ammonia vapor appear as fine, shallow lines or fissures, and are best detected with the aid of a microscope. The types of cracking that occurred are illustrated in Figure 3.

All specimens exposed to ammonia vapor developed a characteristic black tarnish.

Amine Vapor Test

All cases, whether annealed or unannealed, did not exhibit stress corrosion cracking after 25 hours contact with vapor of any of the amines. Consequently, the tests were discontinued.

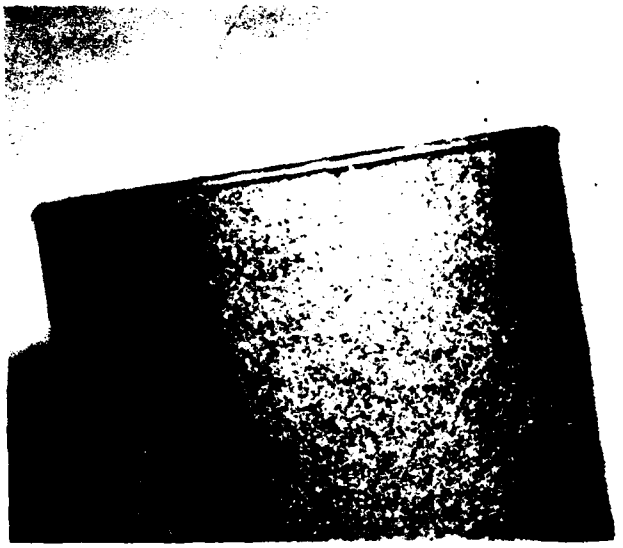
No tarnish was evident on any of the specimens.



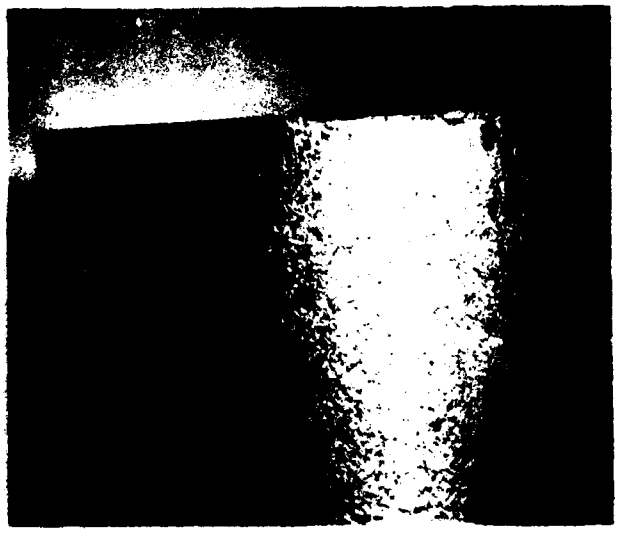
Annealed (Stress Relieved)

Unannealed

Figure 2. Mercurous Nitrate Test (30 Minutes) - 15X



Annealed (Stress Relieved)



Unannealed

Figure 3. Ammonia (8.06M) Vapor Test (30 Minutes) - 15X

TABLE I.

Stress Corrosion Cracking in Ammonium Hydroxide Vapor-
Unannealed Specimens

Solution	Exposure Time (minutes) 15	20	30
NH_4OH - 8.06 M (30% w)	R	R	RN
NH_4OH - 7.25 M (27% w)	R	RN	RN
NH_4OH - 6.44 M (24% w)	R	R	R
NH_4OH - 5.64 M (21% w)	R	R	R
NH_4OH - 4.83 M (18% w)	R	R	R
NH_4OH - 4.03 M (15% w)	0	R	R
NH_4OH - 3.22 M (12% w)	0	0	R
NH_4OH - 2.42 M (9% w)	0	0	R
NH_4OH - 1.61 M (6% w)	0	R	R
NH_4OH - 0.81 (3% w)	0	0	R
Control - H_2O	0	0	0

NOTE: 0 = No cracks
R = Fine cracks around rim.
RN = Fine cracks around rim and neck

TABLE II.

Stress Corrosion Cracking in Ammonium Hydroxide Vapor-Annealed Specimens

Solution	Exposure Time (minutes) 15	20	30
NH_4OH - 8.06 M (30% w)	R	R	R
NH_4OH - 7.25 M (27% w)	R	R	R
NH_4OH - 6.44 M (24% w)	R	R	R
NH_4OH - 5.64 M (21% w)	R	R	R
NH_4OH - 4.83 M (18 % w)	0	0	R
NH_4OH - 4.03 M (15% w)	0	0	R
NH_4OH - 3.22 M (12% w)	0	0	R
NH_4OH - 2.42 M (9% w)	0	0	R
NH_4OH - 1.61 M (6% w)	0	0	0
NH_4OH - 0.81 M (3% w)	0	0	0
Control - H_2O	0	0	0

NOTE: 0 = No cracks.
R = Fine cracks around rim.

Amine Immersion Test

Stress corrosion cracking did not occur in annealed or unannealed cases immersed in each of the amine liquids after 24 hours. The specimens did not develop a tarnish film.

Ammonium Hydroxide Immersion Test

Specimens failed to exhibit stress corrosion cracking after 48 hours in ammonium hydroxide (30 percent/weight). No tarnish film was evident.

Ammonium Hydroxide and Hydrogen Peroxide Immersion Test

Specimens developed a black tarnish but failed to show any evidence of cracks after one hour exposure. The tarnish consisted of isolated patches around the cases.

Ammonium Carbonate Test

Specimens did not develop stress corrosion cracks after a 90-minute exposure period in the ammonia-carbon dioxide environment. Tarnish did not develop on any of these specimens.

Mattsson's Solution

The results of the Mattsson's solution test are summarized in Table III. All test specimens developed a black film, and copper sulfate crystals were attached to specimens exposed over one hour. Results which are typical of those occurring in the Mattsson's solution for the annealed or unannealed cases are illustrated in Figure 4.

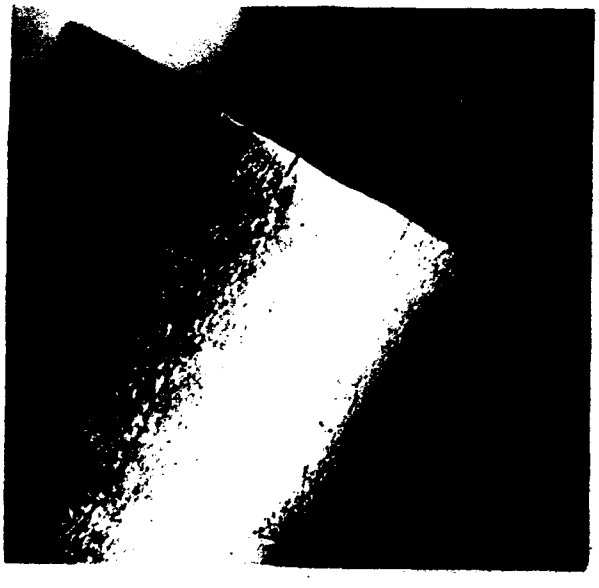
TABLE III.

Susceptibility of Annealed and Unannealed Cartridge Cases In Mattsson's Solution

Time (hrs)	Annealed	Unannealed
1	0	R
2	0	RS
3	0	R

0 = No cracks.
R = Fine cracks around rim.
RS = Fine cracks around shoulder.

Annealed (Stress Relieved)



Unannealed

Figure 4. Mattsson Test (120 Minutes) - 15X

Ammonium Vapor and Anodic Current Test

All unannealed and annealed cases exhibited fine cracks around the rim when subjected to an anodic potential of either 3 or 6 volts. Cracks formed on the annealed specimens were perceptively finer. The effects of this test on annealed and unannealed specimens are illustrated in Figure 5. In essence this observation coincides with the results of specimens subjected to ammonia vapor in the absence of imposed anodic potential. On the other hand, marked dissolution of the rim edges of specimens of both groups occurred at 6 volts. Cracking was not accelerated nor intensified under conditions of current flow, in each situation, in ammonia vapor environment.

DISCUSSION

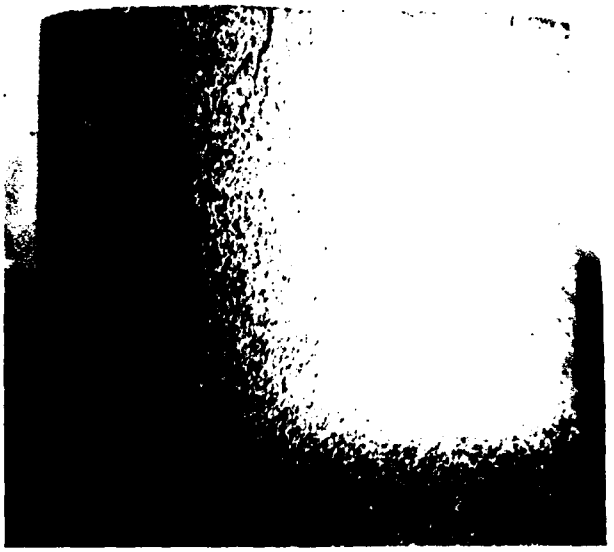
Stress corrosion cracking of the brass cases occurred in several of the test environments employed. It was noted that the depth or extent of cracking differed with the environment test method employed.

Cracking of stressed brass cases which results from the amalgamation of mercury is quite severe. Penetration through the case wall is characteristic. It should be noted that all cracking in the mercurous nitrate test was confined to unannealed specimens. In no case did cracking occur with annealed specimens.

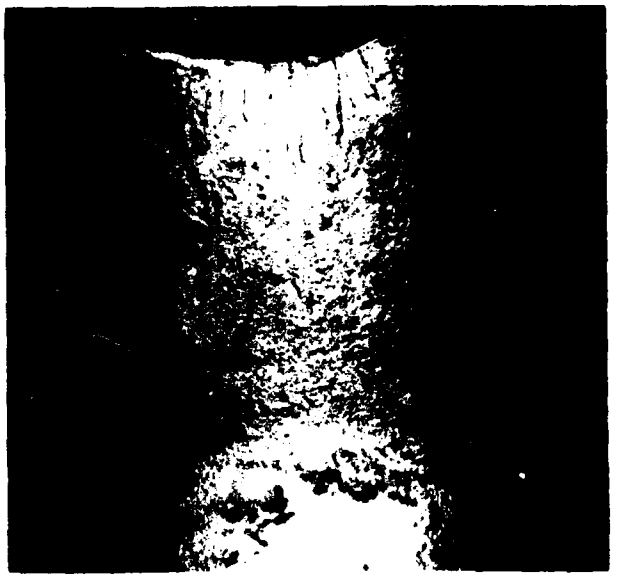
Unlike the distinct results of the mercurous nitrate test (cracking vs no cracking), the stress corrosion cracking resulting from ammonia vapor is subject to interpretation. Both stressed and annealed specimens undergo stress corrosion cracking in ammonia vapor. However, the cracks are so similar that it is difficult to tell whether the test specimen is annealed or not. The cracks occurred in the neck and rim of the cartridge case and did not penetrate the entire thickness of the specimen. All incidences of cracking in ammoniacal environments are accompanied by a black tarnish over the entire cartridge case.

It is indicated that the ammonia causes cracks in stressed brass at lower stress levels than does mercurous nitrate solution. A study to determine the tensile stress threshold of brass cases subjected to ammonia vapor might be of value.

Vapor tests utilizing the various amines produced no cracks in stressed specimens over the exposure periods employed. The specimens



Annealed (At 3 volts)



Unannealed (At 6 volts)

Figure 5. Anodic Current-Ammonia Vapor Test (20 Minutes) - 15X

were exposed for periods up to 25 hours. Rosenthal and Jamieson[3] reported that in triethanolamine cracking did not occur until after 45 days' exposure. In contrast, cracking in ethylamine resulted more rapidly, i.e., in three days time. Consequently, amines must be discounted in considering a rapid, effective means for indicating stress corrosion susceptibility of brass cases.

Immersion tests utilizing ammonium hydroxide, diethylamine, 2-ethylhexylamine and cetyl dimethylamine failed to result in cracking. In all of these immersion trials, tarnish film failed to develop. It would appear that a surface film is necessary for the initiation of cracking. The importance of tarnish film in the cracking of brass has been upheld by Shimodaira and Takano.[6] This requirement of a surface film was also indicated from the ammonia vapor tests performed in this investigation. If no tarnish formed, as was experienced when dry specimens were used in the ammonium carbonate vapor test, no cracking occurred.

It was thought that additions of hydrogen peroxide to ammonium hydroxide might assist in producing a tarnish film, and enhance stress corrosion cracking in the specimens. The fact that ammonium hydroxide and hydrogen peroxide did not promote cracking may be attributed to the uneven oxide that developed on the specimens. The reaction between brass and ammonia appears to be dependent on a uniform film formed on the specimen. The precise mechanism is not understood, but evidence that such conditions initiate cracking is widely documented.

The effect of ammonia vapor concentration on the time until cracking does not appear to be a critical factor. Vapors from solutions ranging from 8.06 M to 0.81 M will cause some degree of cracking in unannealed specimens within 30 minutes. The annealed cases exhibited cracking in concentrations ranging from 8.06 to 2.42 M within 30 minutes. It seems unlikely that a test could be developed based on concentration differences. Again, some means of differentiating the extent of cracking would have to be devised, since differences between cracks developed in annealed and unannealed cases are not obvious, even under low power magnification (ca. 50x).

The cartridge cases failed to crack in the ammonium carbonate test. Also evident was the lack of tarnish film. It is assumed that carbon dioxide inhibited the surface reaction that normally would

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[3] H. Rosenthal and A. L. Jamieson Trans. AIME, 156, 212 (1944).

[6] S. Shimodaira and M. Takano, Fundamental Aspects of Stress Corrosion Cracking, Proceedings of Conference, September 11-15, 1967, NACE, Houston, Texas, 1969, p. 202.

occur in ammonia vapor as was indicated, but not fully confirmed, by Johnston.[7] Wetting the ammonium carbonate did not alter this result.

Electrochemical aspects of stress corrosion cracking of brass and the influences of applied anodic potential, as offered by several researchers, are summarized by Bailey.[8] Under suitable conditions, stressed brass in an appropriate electrolyte solution and subjected to applied anodic potential will exhibit cracking propensity. Hoar and Booker[9] investigated the effects of anodic potential on the cracking of brass in solution, and reported that at a current density of 835 $\mu a/cm^2$ the cracking time was reduced.

Information was not available concerning the influence of anodic polarization on the cracking of brass in ammonia vapor. In this investigation an effort was made to acquire some indication regarding this aspect. This approach was considered to have some merit because, as indicated above, stress corrosion cracking of brass in ammonia vapor is associated with the existence of a thin film of electrolyte in the surface of the brass.

In these experiments the concentration of the electrolyte film on the specimens was not established. Further, the current density over the film (or specimen) was considered highly non-uniform, and that the maximum current density was in the mouth and neck region in contact with the felt wick. (Based on the total area of the cartridge case, 29.8 cm^2, and assuming uniform current distribution over the surface, the current density at 3 volts would be ca. 27 ma/cm^2, and at 6 volts, 104 ma/cm^2.)

Anodic polarization, as performed, did not result in enhancement of stress corrosion cracking of the brass cases, whether unannealed or annealed, but served only to promote dissolution of the cases at areas adjacent and in contact with the electrolyte solution.

In the Mattsson test, cracking of the unannealed cases occurred in about 120 minutes, whereas in the mercurous solution cracking was accomplished in about 60 minutes, including time for vaporizing mercury from the specimens. The Mattsson's solution produced fine cracks in the neck and shoulder of the case, but the mercurous solution resulted in through-wall cracks in the same regions. Each solution produced cracks only in the unannealed specimens. Significantly then, either

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[7] R. G. Johnston, Sheet Metal Ind., 1940, 14, p. 1197.

[8] A. R. Bailey, Met. Rev., 6, 101, (1961), p. 114.

[9] T. P. Hoar and C. J. L. Booker, Corrosion Science, 5, 821, (1965).

test serves to discriminate between unannealed and annealed cases via the presence of cracks or the absence of cracks respectively. The degree of cracking that occurred in Mattsson's solution was similar to that produced in ammonia vapor (Figure 2). The cracks were fine and did not penetrate the case wall. Unlike those developed in the mercurous nitrate solution, the cracks resulting in the Mattsson's solution were not readily observable without the aid of a microscope.

The ease with which the two tests are executed is not a critical factor in their comparison. The preparation of the Mattsson's solution is rather cumbersone because it involves such large quantities of solution. A common criticism is that all of the copper sulfate fails to go into solution. On the other hand, the mercurous nitrate test entails high temperature and hood facilities for the removal of amalgamated mercury. With respect to the total time involved for completing each test, the Mattsson procedure requires about double the time for the mercurous nitrate test. Thus as a rapid test, the Mattsson test potentially is of use and is adaptable. This aspect along with previously considered advantages, i.e., differentiation between unannealed and annealed cartridge cases and freedom from pollution and toxicity, support the Mattsson method as a substitute for the mercurous nitrate test method.

CONCLUSIONS

The ammonia vapor test method for determining the susceptibility of brass cartridge cases to stress corrosion cracking is not suitable as a standard quality-control method. In a short-term period the method does not differentiate, either by type or pattern of cracks which develop, between annealed (relieved) and unannealed (stressed) cases.

The failure of stressed brass in Mattsson solution is easily detected in short time, and it appears that this immersion method is an attractive alternative to the mercurous nitrate test. The good reproducibility, and the non-toxic and non-polluting character of the method, favor its usefulness as a quality-control method.

RECOMMENDATION

Further investigations are recommended toward accomplishing modifications to the Mattsson method for the purpose of accelerating initiation and accentuation of cracking in susceptible brass cartridge cases, and for ascertaining its potential as a substitute for the standard mercurous nitrate test.

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6. S. Shimodaira and M. Takano, Fundamental Aspects of Stress Corrosion Cracking, Proceedings of Conference, September 11-15, 1967, NACE, Houston, Texas, 1969, p. 202.

7. R. G. Johnston, Sheet Metal Ind., 1940, 14, p. 1197.

8. A. R. Bailey, Met. Rev., 6, 101, (1961), p. 114.

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